

### ONR FINAL TECHNICAL REPORT

A DoD University Research Initiatives Center Office of Naval Research, Arlington, VA

Professor James Economy Principal Investigator

March, 1992

Summary of Research
5 Year Project Period
15 September 1986 to 30 December 1991
Contract #N00014-86-0799



Approved for public released
Distribution Unlimited

93-01360<sub>N</sub>0

# ONR Final Technical Report

# National Center for Composite Materials Research at University of Illinois, Urbana - Champaign

# **Table of Contents**

Item	Page #
Overview of Program	1
ONR Data Base Information	2
Significant Results	
1. Materials Design and Selection	4
2. Processing of Thick Section Composites	10
3. Mechanical Behavior of Thick Section Compos	ites 14
Other Sponsored Research	24
Listing of Publications	26
Listing of Technical Reports	34
Student Degree List	38
Faculty / Postdoctoral Data	39
Subcontracting Universities - Technical Report Summ	naries 40
;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;;	By
STATEMENT A PER TELECON OR ROSHDY BARSOUM ONR/1132 ARLINGTON, VA 22217-5000	Availability Codes  Dist Avail and for Special

# ONR FINAL TECHNICAL REPORT National Center for Composite Materials Research University of Illinois - ONR Contract #N00014-86-K-0799

### Summary of Research

# Description of the scientific research goals

A key objective of the ONR-URI Center is to conduct basic research on critical materials science, mechanics, and structural problems currently confronting the Navy and to be encountered in its future use of advanced composite materials. The current emphasis of the Center's research thrust is placed on thick-section structural composites and composite structures, since these subjects have been identified as major potential problem areas of concern in the present and future high-performance Navy structural systems.

The research program had three major thrusts, namely:

- 1) Materials design and selection.
- 2) Processing of thick filament wound cylinders.
- 3) Mechanical behavior of thick section composites.

# Areas that were emphasized include:

- a). A broad based program on new and improved matrices, interfaces and reinforcing agents which involves the efforts of three faculty (J. Economy, thrust leader (acting)).
- b). A team effort by four faculty on processing of thick section composites (C. Tucker, thrust leader).
- c). A coordinated effort at the micromechanical, macromechanical and structural scale involving the efforts of seven faculty (D. Socie, thrust leader).

### **ONR** Data Base Information

R&T Number: p400013uf109 (ONR contract# N00014-86-K-0799)

Contract/Grant Title: A Proposal for the Establishment of a Center

for Advanced Composite Materials Research

Scientific Officer: Dr. Roshdy S. Barsoum

Principal Investigator: Professor James Economy

Mailing Address: Department of Materials Science and Engineering

University of Illinois
1304 West Green Street

<u>Urbana</u>. IL 61801

Phone Number: (217) 333-1441

FAX Number: (217) 333-2736

E-Mail Address: none

a. Number of papers submitted to referred journal but not yet published: 15

b. Number of papers published in referred journals: (list attached) 31

- c. Number of books or chapters submitted but not yet published: none
- d. Number of books or chapters published (list attached): 3
- e. Number of printed technical reports & non-referred papers (list attached): 68
- f. Number of patents filed: none
- g. Number of patents granted: none
- h. Number of invited presentations at workshops or professional society meetings (list attached): 21

- i. Number of presentations at workshops or professional society meetings (list attached): none
- j. Honors/Awards/Prizes for contract/grant employees: (see list below) may include society awards/offices, promotions, faculty awards/offices, etc.) 3
- k. Providing the following information will assist with statistical purposes.

PI/CO-PI:	Total Female Minority*	23 2 0	Grad	Students:*	*Total Female Minority*	36 _3 1
			Post	Doc:**	Total Female Minority*	9 1 0

- I. Degrees Granted (list Attached):
- \* Underrepresented or minority groups include Blacks, Hispanics, and Native Americans. Asians are not considered an underrepresented or minority group in science and engineering.
- \*\* Supported at least 25% this year on contract/grant.
- 5. Patents granted: none
- 6. Degrees granted: (see attached list)

# LIST OF AWARDS/HONORS/PRIZES

Name of Person Receiving Award	Recipient's Institution	Name of Award	Sponsor of Award
D. Dean (Stdnt)	U. of Illinois	Harris Fellow	Harris Found
I.D. Parsons (Prof)	U. of Illinois	PYI	NSF
T. Twardowski (Stdnt)	U. of Illinois	Best Stu Paper	Am Soc Comp

### MATERIALS DESIGN AND SELECTION

The materials effort at the Center during the funding period involved four general areas, graphite-epoxy composites, thermoplastic matrices, liquid crystal polymers as matrices and coupling agents, and more recently ceramic-metal composites consisting of aluminum diboride flakes in an aluminum matrix. The work was performed in the laboratories of three principal investigators, P. H. Geil (graphite-epoxy, thermoplastics), S. I. Stupp (liquid crystal polymers), and J. Economy (AlB<sub>2</sub> - Al). The program thus covered research on materials in use at the present time (epoxy-based), materials at the experiment/production interface (thermoplastics) and future materials (liquid crystal polymers and boride-metal composites).

# I. <u>Graphite - Epoxy Systems. P.H. Geil</u> <u>Thick Section Curing - Computer Simulation and Experimental</u> <u>Measurements</u>

Thick part (>5 cm) temperature profiles have been obtained from a ID experiment and compared with the results of a computer simulation containing new and existing models for extent of reaction, viscosity and composite properties. The simulation recovers the thermal behavior of a 5 cm thick, unidirectional Hercules AS-4/3501-6 graphite/epoxy composite part. Application of consolidation pressures based on predicted viscosities led to dramatic improvement in void content of a real part. The simulation was used to investigate important processing variables and changes in chemorheology and temperature in a thick section part. The following results were found: initial extent of reaction is relatively unimportant: consolidation is important: the peak temperature originates near surface of part and propagates to the center; asymmetric temperature application changes profiles; viscosity never reaches low values simultaneously through the thickness in parts in excess of 10 cm suggesting single stage processing is impractical for samples of such thicknesses.

# II. <u>Thermoplastics</u>, P.H. <u>Geil</u> Physical Aging and Its Effect on Long Term Creep. Stress Relaxation and Fracture Toughness

Long term creep is always listed as a potential detrimental problem in elevated temperature applications of composites with thermoplastic polymer matrices; in crystalline polymers as used above Tg and in amorphous polymers as the use temperature approaches Tg. In previous work in the Center, A. Miyase showed near cessation of creep in composites based on the crystalline polymer JI in the viscoplastic load range at temperatures near but above Tg. We have shown similar effects in composites based on the amorphous polymer Torlon near but below Tg. In the latter case the shrinkage and increase in stiffness due to physical aging was shown to "compensate" for the creep due to the applied load. Similar studies of long term creep, recoverable creep and changes in physical properties due to aging above the nominal Tg, have also been completed on JI and JI/AS4 composite at and above Tg. JI is semicrystalline; the aging in this case is attributed to the presence of a distributed or double Tg. The studies of Torlon were also extended to examine the concurrent effect of physical aging and stress relaxation and the effect of physical aging on fracture toughness at elevated temperatures. The results to date suggest long term creep in tension should not be a problem in thermoplastic resin composites, the molecular motions involved in physical aging compensating for those occurring in creep; in compression, however, the two effects would have the same sign. In addition the results clearly demonstrate the inadequacy of time-temperature superposition for predicting long term physical properties.

### Characterization of Jl and Jl /A54 Composites

JI/AS4 composite has been utilized in the Center as a generic thermoplastic matrix composite - for materials science, mechanics and structural research. The materials characterization has been completed. Results include: a) X-ray diffraction and DSC studies have shown complex, temperature dependent, reversible and irreversible changes in the crystal structure of the neat resin with accompanying changes in physical properties of both resin and composite. In particular annealing at 240°C results in an

irreversible crystal-crystal phase change and a significant improvement in properties and, b) modeling of moisture diffusion into JI composites has been successful permitting correlation with experimental measurements of moisture diffusion and the accompanying changes in physical properties.

# III. Liquid Crystal Polymers, S. Stupp Main Chain Nematic Polymers

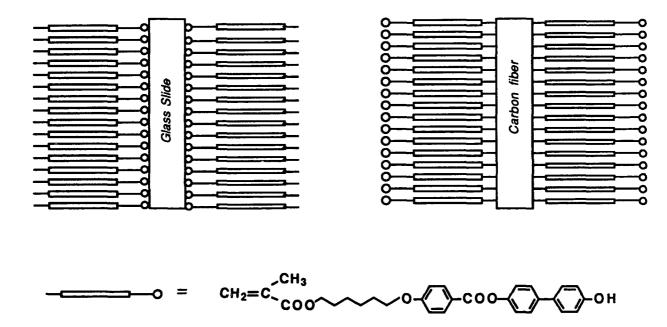
In the early part of the program we investigated a model system consisting of a main chain nematic polyester as a matrix surrounding carbon fibers. The first phase of this work involved studies of molecular orientation in the matrix as affected by the presence of carbon fibers. Using microscopy and broadline NMR measurements we discovered that fibers created long range orientation of polymer molecules around them. A consequence of this phenomenon was a faster rate of macroscopic orientation in the matrix under an external force in the presence of dispersed fibers. In the second phase of this work we demonstrated the feasibility of chemically grafting these nematic polymers to carbon fibers. was accomplished through a methodology in which monomers were polymerized in the presence of oxidized fibers. Electron microscopy demonstrated excellent wetting at the fiber-matrix interface in the grafted fibers. In great contrast model composites containing the same fibers and matrix revealed extremely poor interfacial bonding.

# Side Chain Liquid Crystal Polymers

Our work on main chain nematic polymers suggested the possibility of using side chain liquid crystal polymers to design molecularly ordered interfaces in advanced composite materials. We were able to synthesize comb-shaped self-ordering polymers in which molecular teeth were functionalized at their termini. These chemical functions could be used in principle to create covalent bonds between organic molecules at the interface and the surfaces of fibers. Furthermore, these same functions may also form bonds with polymeric materials used as composite matrices thus opening the door to the concept of self-ordering coupling agents.

During this past year we investigated the possibility of converting the functionalized side chain liquid crystal polymer into

Langmuir-Blodgett (LB) thin films to create monolayers of molecules on carbon fiber surfaces. The unique properties of these LB films were used to form highly ordered monomolecular layers of coupling agents at carbon fiber-matrix interfaces and the properties of the resulting composites were studied. The polymer investigated was the liquid crystalline amphiphile synthesized and studied in previous years of the project which has the potential to form ordered monolayers on both polar and non-polar substrates such as glass slides and carbon fibers. The possible structures are shown below.



The functionalized carbon fibers (AS4 12K, Hercules Inc.) were used as received. Transfer of LB films of the polymer on functionalized carbon fibers was tested with various bases and after dipping the fibers were dried in vacuo overnight at 50°C. Measurement of contact angles on fibers verified the effectiveness of the LB transfer. The measurements also revealed that layers were most likely transferred with phenolic functional groups exposed on the outer surface. For the fibers coated with polymer, contact angle measurements indicated a small contact angle for water due to the high hydrophilicity of the phenolic groups. These groups are capable of reacting with epoxide groups of the matrix-forming monomer.

Single fiber tests were then carried out on dipped fibers encapsulated in a cured epoxy matrix. A striking result was observed, namely, the permanent birefringence obtained with coated fibers is 1.8 times greater than the one recorded with the control fibers. This suggests that the strength of interfacial adhesion is very much increased by the presence of a single molecular layer of polymer at the fiber/matrix interface.

It can be concluded that the fibers coated with a film of monomolecular thickness show a clear improvement in load transfer at the fiber/matrix interface relative to controls. Stress birefringence revealed pronounced plastic deformation in the matrix caused by a strong interfacial adhesion. Furthermore, critical length data from single fiber tests revealed an increase in interfacial shear strength tc. This improvement in tc is probably due to covalent bonds formed between the phenolic groups of the polymer side chains and the oxirane rings of the matrix and also to highly ordered boundary zones around the fibers. This improvement in interfacial adhesion is even clearer than in fibers that were chemically grafted with the same polymer. As far as we know our results are the first to demonstrate a direct link between a well defined molecular scale modification of a fiber surface and macroscopic properties of a composite system.

# IV. Boride - Al Systems, J. Economy

The goal of this project was to prepare single crystal flakes of AlB<sub>2</sub> which could greatly enhance the compressive properties of ternary composites consisting of alternating plies of graphite fibers and AlB<sub>2</sub> flakes in an epoxy matrix and still permit for high directional properties. Single crystal flakes of AlB<sub>2</sub> measuring a few microns in thickness and several mm in width have been shown to provide excellent compressive properties in an epoxy matrix. In fact such composites display flexural, tensile and compressive modulus values in the plane comparable to what one observes with high modulus graphite fibers in one direction. One problem with these composites has been the presence of cubic crystals of a-AlB<sub>2</sub> which greatly compromise the strength properties. It would be highly desirable to develop a synthetic route which uniquely yields the AlB<sub>2</sub> crystals.

Major progress was made in setting up a high temperature reactor for crystallizing AlB<sub>2</sub> flakes in molten aluminum. A furnace has now been developed to produce AlB<sub>2</sub> flakes dispersed in an aluminum matrix through a peritectic reaction which would avoid formation of a-AlBl<sub>2</sub>. The method that is being utilized involves the bubbling of boron trichloride through molten aluminum at a temperature just below the peritectic point. Also, an apparatus is being devised to produce the flakes in an aluminum matrix in order to optimize the size and volume fraction of the flakes at various reaction temperatures, cooling rates, and reaction times. This second apparatus has been designed and is expected to be built by the end of February.

### Mechanics of Processing of Composite Materials

The manufacture of thick composite sections poses unique processing problems which have not been addressed in conventional composites manufacturing. For thick section parts the low thermal conductivity of the polymer matrix leads to thermal lagging in the heating phase of the process cycle, and thermal spiking during the exothermic cure between interior temperatures and skin or autoclave temperatures. High residual stresses can lead to matrix micro cracking and transverse cracking during the cooldown phase of the process cycle. The utilization of prepreg tapes in the lav-up or filament winding of thick sections can cause non uniform consolidation through the thickness. Additionally, the manufacturing time for thick sections utilizing conventional manufacturing techniques is estimated to be quite long.

These issues were addressed through a coordinated group of projects. The individual topics included:

- assessment of conventional curing methods for very thick sections
- exploration of staged curing strategies
- modeling of residual stresses in filament winding
- · development and examination of a continuous curing process

# Conventional Curing of Thick Sections, P.H. Geil

Numerical models for the temperature and cure histories in autoclave-type curing were tested against experiments on thick sections of glass-epoxy and carbon-epoxy composites. The experiments involved 200-ply laminates with embedded thermocouples. The results show that the model must include the influence of consolidation on the thermal properties of the composite to achieve quantitative agreement with the experiments. The manufacturer's recommended cure schedules for "thick" sections gave better control of the reaction exotherm than the thin-section curing schedules. However, the calculations reveal that thicker sections (10 cm and up) cannot be cured satisfactorily using conventional techniques. In these very thick sections the outer layers gel before the center of the laminate has become warm; there is no time at which the resin is fluid across the entire laminate, so there is no opportunity to consolidate the part.

# PROCESSING (continued)

### Staged Curing, S. White

To address this issue a modified manufacturing technique called stage-curing was investigated. In this technique a certain thickness of material is wound (filament winding) or layed-up (hand lay-up) and subjected to a partial cure in which gelation is induced. The incremental thickness is also consolidated during this step. Another incremental thickness is then applied on top of the first, and the assembly is again subjected to a partial cure to gelation. In this fashion the part thickness is increased in stages until the final thickness is achieved. Afterwards, the entire structure is subjected to a finishing cure cycle. Structures manufactured by staged curing are uniformly cured and posses uniform fiber volume distribution through the thickness.

Staged cured specimens of two polymer composite systems were optically examined for process quality and tested for mechanical integrity utilizing the DCB and interlaminar shear tests. Preliminary studies on a BMI matrix system (BASF 5250-2) indicated a significant reduction in DCB mode-I interlaminar fracture toughness for stage curing. Subsequent testing of a graphite/epoxy system (AS4/3501-6) led to the same conclusions. However, microscopic analysis of these specimens showed significant void production for stage cured specimens while voidfree laminates were achieved for conventional curing. Subsequently, two approaches were utilized to correct this problem. In the first. several cure cycle modifications were investigated to suppress void production in the stage curing of laminates. It was found that increased consolidation pressure together with recycled stage one processing effectively suppressed void production for stage curing. Secondly, interlaminar shear specimens were manufactured for both stage curing and conventional curing. The interlaminar shear test is relatively insensitive to small variations in void content between the two groups of specimens. Mechanical testing of these specimens yielded results which indicated no sacrifice in interstage bonding.

# PROCESSING (continued)

### Residual Stresses, S. White

Research was also carried out on the modeling of processing-induced residual stresses in filament wound composites. The model is thermoelastic and accounts for chemical and thermal deformations during cure and allows for cure dependence of mechanical properties. The thermal distribution is solved using an explicit marching scheme and the equilibrium equations are solved using generalized plane strain finite elements. This model was used to investigate the influence of several processing and material parameters on the residual stress distribution.

The influence of mandrel material was investigated by varying mandrel stiffness and thermal expansion. For high stiffness and low thermal expansion materials, the separation time during cooldown is much shorter. This significantly effects the final residual stress profile. For an aluminum mandrel the maximum hoop residual stresses are located next to the mandrel surface and were calculated to be 25 MPa. For a steel mandrel the maximum is now located on the outer surface of the composite tube and is reduced in magnitude to 15 MPa. As the chemical shrinkage of the matrix material is increased the radial and hoop stresses become increasingly critical. The maximum hoop stress at the inner surface of the composite increases by a factor of two for a volumetric chemical shrinkage of 5%.

# Continuous Curing, S. White and C. Tucker

Another strategy for making very thick sections is continuous curing. Composite pre-preg is wound or laid up continuously onto a heated mandrel or tool. When the process is managed properly, a cure front propagates out from the tool at the same speed that the thickness of the composite is growing. The cure front stays a fixed distance below the surface, and consolidation can take place in this uncured layer. Thermal analysis shows that staged curing can be accomplished without any over-temperature in the composite, regardless of thickness. Also, the cured composite is held at a uniform temperature, minimizing thermal stresses during the lay-up or winding stage. This is a contrast to current "cure-on-the-fly" approaches, which heat the pre-preg as it is laid down.

### PROCESSING (continued)

Both modeling and experiments have been used to study the continuous curing process. We have identified certain conditions under which the curing process is unstable, and have shown experimentally that thick sections can be cured this way. This process deserves further study as an approach to making very thick composites with thermoset matrices.

### Summary, C. Tucker

In summary, conventional curing methods are completely unsuitable for very thick section composites. The use of stage curing for thick section composites was shown to be feasible, in that no mechanical degradation of the bondline between staged sections was found. Low void content specimens were produced using AS4/3501-6 with stage one corresponding to the initial consolidation dwell of the manufacturer's recommended cure cycle. For filament winding of thick section composites the use of a low chemical shrinkage resin is advantageous for reduction of processing-induced residual stresses and residual stress distributions can be controlled through the proper choice of mandrel materials. A new processing approach, continuous curing, offers a way to overcome the thermal limitations in the manufacture of very thick sections

### Mechanical Behavior of Composite Laminates

### Summary

The mechanical behavior of composite materials have been studied at three different levels: micromechanical, macromechanical and structural scales. Micromechanical studies included a study of the glass transition temperature changes at the interphase between the fiber and matrix. Micromechanical modeling and microstructural analysis was used to study the failure mechanisms of carbon fiber laminates under compressive deformation. Biaxial compression tests were conducted on laminates to determine the failure modes and failure mode dependence for multiaxial loading conditions. Structural analysis consisted of a study of the buckling behavior of stiffened shells and the development of a material model for nonlinear structural analysis. A short summary of the various research projects is presented.

### **Micromechanical**

The Influence of the Fiber/Matrix Interface on Local Glass Transition Temperatures in Composites, N.R. Sottos

Local structure and properties of the interphase is crucial to understanding how the interphase influences the overall thermomechanical behavior of a composite laminate. theoretical investigations present evidence for the existence of an interphase region with a glass transition temperature that is significantly lower than that of the neat resin. In the current study both interferometric measurements and single fiber critical length tests were performed on samples with tailored interphases to further investigate variations in local glass transition temperature and its influence on micromechanical behavior. Three different interphase conditions were considered: fibers coated with a low To resin (EPON 871), fibers coated with a high T<sub>a</sub> resin (EPON 164), and untreated fibers (no coating). Experimental results support the existence of a reduced glass transition temperature near the fiber Additionally, the significant influence of the interphase  $T_{\alpha}$ on thermally induced microcracking was demonstrated.

# Compressive Fatigue of Carbon Fiber Reinforced Polymer-Matrix Composites, J.K. Shang

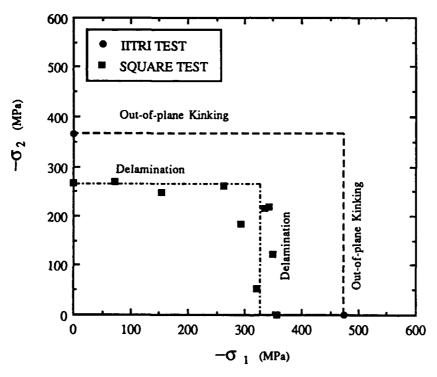
A preliminary study on the compressive fatigue behavior was conducted in carbon fiber reinforced epoxy composites with ply orientations of 0°, (+/- 45°)s and (90°)s under uniaxial compression and pure bending conditions. The primary objective of the research was to understand the relationship between the compressive strength of the composite and the properties of the composite constituents. It was found that the compressive strength of carbon fiber composite is adversely affected by constant amplitude fatigue loading. The loss in compressive strength due to constant amplitude fatigue depends on fiber orientation; for three fiber directions, 0°, +/-45° and 90°, 0° composite suffers the most strength reduction. Examinations of the failure mechanism revealed that interfacial failure, which takes the form of axial cracking in 0° composite, delamination in +/-45° and fiber/matrix bebonding in 90° specimen, is the primary damage mechanism under compressive fatigue. In the 0° composite, it was observed for the first time that the composite exhibits cyclic strengthening behavior following step loading. compressive strength and the compressive modulus of the composite are shown to increase with compressive fatigue loading. physical basis for the cyclic strengthening behavior is not clear at this time but appears to be the compaction of polymer molecules upon unloading following the matrix plastic deformation.

### **Macromechanical**

# Biaxial Compressive Behavior of Composite Laminates. D.F. Socie

A test system for evaluating in-plane biaxial compressive deformation was developed. A special test fixture was used to apply biaxial loads. These tests were conducted in a test frame that has four independent liner actuators arranged in a cruciform shape. Alignment of the loading axis is critical for conducting compression tests. Flexible end reinforcements were used to transfer the external loads to a square specimen where the entire specimen is subjected to a uniform compressive load. The flexible reinforcement is stiff only in the loading direction. Compressive strengths from this new specimen are considerably lower than those

obtained from standard IITRI specimens. This difference is due to a change in failure mode. A strength and failure mechanism study of E-Glass/Epoxy laminates subjected to uniaxial and in-plane biaxial Specimen configuration was compression loadings was conducted. found to have a large influence on the damage mechanism and resulting strength of the laminates. Standard compression test specimens with a gage section of 12.5 mm fail in a shear damage or a kink band mode. In large 125 mm square specimens, significant delamination precedes shear failures and reduces the failure strength by 20-35%. The biaxial compressive failure envelope for E-Glass/Epoxy is adequately described by a maximum stress or a maximum strain criteria that considers the different failure modes. Estimates of the failure envelope based on small compression test specimens where the failure mode is a shear or a kink band overestimates the strength of the large square biaxial test specimens.



Failure Envelope of Cross-Ply Laminates

The circular data points along the axis represent standard IITRI tests. Biaxial test data is shown with square datapoints.

### Delamination growth in Biaxial Compression, D.F. Socie

Delamination growth in laminated composites was investigated under biaxial compressive stresses. The critical stress to initiate and grow a pre-existing delamination was calculated using an energy criteria and fracture mechanics. An expression was derived for this critical stress as a function of the material properties and size of the delamination. Delaminations were introduced in the specimens during manufacture by placing Teflon tapes between plies. Tests were then conducted on unidirectional and cross ply E-Glass/Epoxy 10 ply laminates and found to be in close agreement with the predicted values. Thick section laminates (50 ply) fail by delamination during normal testing. Introduction of additional delaminations did not reduce the strength.

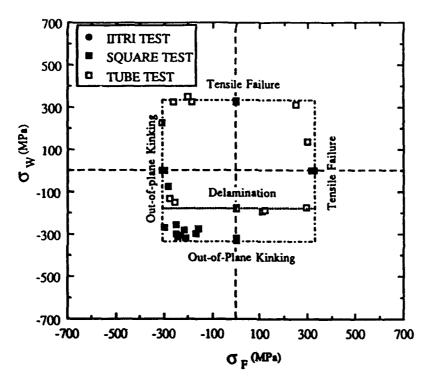
# <u>Failure Criteria for Cutouts under Biaxial Compression Loading. D.F. Socie</u>

An experimental investigation was conducted to study the failure strength and damage mechanisms of notched composite laminates under biaxial compression loading. Square specimens (125 mm) were made from ASTM G-10 orthotropic woven composite laminates by drilling 20 mm diameter center holes. Flexible end reinforcements were employed to prevent edge failure and lateral supports were used to prevent structural buckling. Under biaxial compression loading, one or two fractures nucleated from the edge of the hole perpendicular to one of the two compression loadings depending on the biaxial loading ratio. Fracture surfaces of failed specimens were observed in an SEM and kinking bands at the notch root were found to control the failure of the specimens under both uniaxial and biaxial loadings. Remote failure stress and strain of notched specimens under uniaxial compression is about 50% of the failure stress and strain obtained from unnotched specimens. Under biaxial compression loading, remote failure stress and strain in one direction are increased linearly with increased external loads in the perpendicular direction. Stress and strain at the notch root were computed numerically and experimentally. The average stress and

strain at the notch root were found to control the formation of kink bands and strength of the laminate. A generalized stress concentration factor based on the average stress criteria was successfully used to estimate the failure strength of the notched laminates under biaxial compression loading.

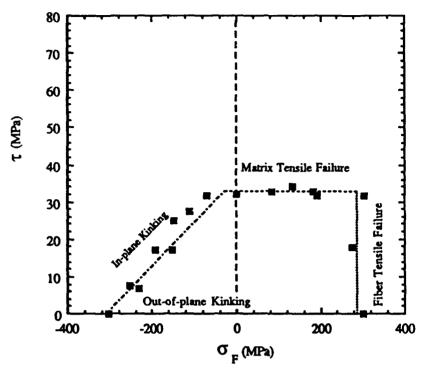
# Failure Mechanisms and Strength of Composite Tubes under Biaxial Loading, D.F. Socie

Failure mechanisms and strength were determined on tubular Loadings consisted of tension, compression, internal pressure, external pressure, torsion and combined loading. These loading conditions allow any combination of in-plane loading to be applied to the specimens. Special test fixtures and extensometers for measuring the deformations during these complex multiaxial loadings were developed. A woven fabric composite, ASTM G-10 was selected for these studies as being representative of a class of composite materials. Warp fibers run parallel to the axis of the tubular specimen. Fill fibers run in the hoop direction. loading produces tensile fiber failures in the fill direction. Compression loading resulted in out-of-plane kink bands. pressure loading of the tubular specimens resulted in delamination Note that the failure modes are independent and the failures. transverse stress does not affect the failure mode or failure strength. As a result the failure envelope is rectangular. Three sets of data are shown for this material. The standard IITRI test results is plotted along the axis and has the same strength as the other specimens. Square plate specimen data is plotted with solid square symbols in the biaxial compression quadrant. Tubular data is plotted with open symbols. Note the difference in compression loading. Flat plate specimens failed in a kink band mode and had strengths that

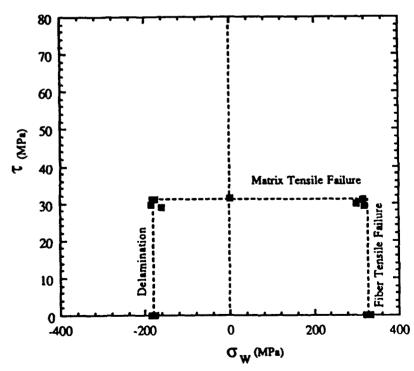


Damage Modes for In-Plane Tension and Compression

were predicted by the standard test. The tubular specimens delaminated under external pressure loading before the kinking mechanism could operate. Fiber tensile failure occurs for tensile Matrix tensile failure occurs for torsion loading. Combined tension and torsion loadings show two independent failure In combined compression and torsion loading in-plane mechanisms. kink bands are formed. Increasing the torsional loads reduces the compressive load carrying capacity of the tubes. This is caused by a rotation of the warp fibers during torsional loading. The fibers rotate as much as 10° during the shear loading. As a result, in-plane kinking is enhanced. Experiments with external pressure and torsion show no interaction between the two loads because the fill fibers (hoop direction) do not rotate during torsion loading. These tests show the importance of considering the failure mechanisms in A complete description for the in-plane strength of biaxial loading. this woven composite has been established. Failure mode independent strength theories, Homman, Fisher, Hill, Tsai-Wu and Tsai-Hill do not reflect the data obtained in these experiments. example, they are incapable of predicting the difference between applying axial compression or external pressure with torsion.



Damage Modes for Axial Loading and Torsion



Damage Modes for Pressure and Torsion

<u>Compressive Deformation and Failure Mechanisms of Unidirectional</u>
<u>Carbon Fiber Reinforced Plastics, H. Sehitoglu</u>

Experimental and theoretical methods were used to characterize the deformation and failure mechanisms of unidirectional carbon fiber reinforced plastics. The experimental program included the manufacture and design of AS4/3501-6 carbon-epoxy composite specimens and the development of experimental apparatus for elevated temperature testing of carbon fiber reinforced plastics. Monotonic tension and compression experiments were conducted on 3501-6 epoxy resin and AS4/3501-6 carbon-epoxy carbon-epoxy composites at temperatures ranging from 25° to 200°C. The tensile and compressive strengths of both materials decrease with increasing temperature. Compressive failure is dominated by longitudinal splitting at temperatures up to 100°C. Above this temperature, the soft matrix allows fiber instability to dominate A three dimensional numerical model was developed to investigate the effects of localized deformation on the compressive deformation behavior of the composite due to thermal and mechanical property mismatch of the constituents, fiber misalignment, and fiber clustering. It was demonstrated that the matrix undergoes localized tensile stresses under compressive loading. The onset of matrix failure is predicted based on local stress-strain behavior.

# Structural

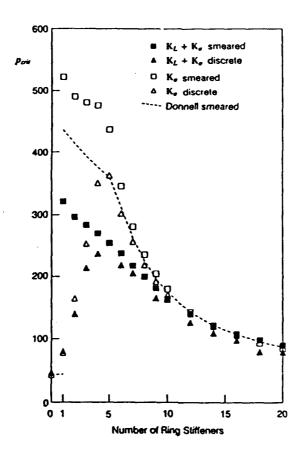
Material Models for Structural Analysis of Thick Section Composites, D. Pecknold

A traditional micromechanical approach which considers the material response on a detailed ply-by-ply basis is not feasible for numerical modeling at the structural or component level to predict overall strength and stability. A new three dimensional nonlinear material modeling approach for thick section composites was developed. The model consists of two main components: a lamina

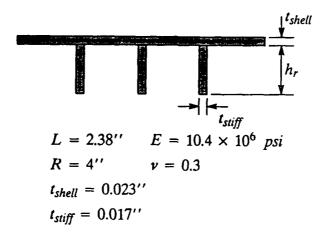
micro-model containing fiber and matrix elements and a simplified unit cell analysis: and a sublaminate model which properly enforces equilibrium of out-of-plane stresses. Evaluations show excellent agreement with detailed numerical micro-modeling of graphite/epoxy laminates. A nonlinear power law material model was incorporated to model matrix softening. Effects of matrix nonlinearity on structural stability were determined.

# The Buckling Behavior of Thick Section, Stiffened Composite Shells, I.D. Parsons

The basic objective of this work was to obtain a detailed understanding of the buckling behavior of stiffened, composite shells. Effects of mode interaction and imperfections have been investigated. The most important result of this research has been an understanding of the effect of orthotropy on the nonlinear prebuckling deformation of shells. This arose through comparisons of different techniques that are commonly used to compute buckling loads of stiffened shells modeled with finite elements. linerized buckling analysis is used where the buckling load is computed by solving a linear eigenvalue problem. Comparisons between the bucking loads with different codes shows a large variation in computed buckling loads. These differences were explained by discovering that cylindrical shells with length to radius ratios less than two are substantially more stiff in one direction (circumferential) than the other (axial) and experience nonlinear prebuckling deformations. Linearized buckling analysis cannot predict the collapse loads of these shells.



Buckling Loads for a Family of Ring Stiffened Shells



Basic Stiffened Shell Configuration.

(The number and depth of the stiffeners were varied by holding all other dimensions fixed, and keeping the ratio of stiffener material to shell wall material in the generator shown constant at 0.24.)

### OTHER SPONSORED RESEARCH

(Include title, sponsor's name, dollar amount and period)

Economy - ONR/DARPA, "Improved Compressives," 10/1/90 - 10/1/92, \$465,000.

<u>Parsons</u> - NSF, "Presidential Young Investigator Award," 9/1/91 through 9/1/96, \$125,000.

<u>Sehitoglu</u> - Brenco Inc., "Micro-Mechanics of Cone Bore Growth," 1/1/91 - 12/31/91, \$43,520.

<u>Sehitoglu & Socie</u> - Mitsubishi Inc., "Mechanisms of Strengthening and High Temperature Properties of Fiber Reinforced Aluminum," 6/1/91 - 5/31/93, \$170,000.

<u>Socie</u> - Caterpillar, "Research in Ceramic Barrier Coatings," no specific time period, \$50,000.

<u>Stubbins</u> - DOE, "Damage Dosimetry & Embrittlement Monitoring of Nuclear Pressure Vessels in Real Time by Magnetic Properties Management" (with John G. Williams and Abderrafi M. Ougouag), 9/30/91 - 9/29/93, \$173,896.

Stubbins - DOE, "Micromechanics & Micromechanisms of Fracture," 1/01/91 - 12/31/91, \$68,368.

<u>Stubbins</u> - UI/CRB, "Center for Multiphase Corrosion/Corrosion in Oil Transmission Facilities," 1/06/91 - 1/05/92, \$18,603.

Stupp - DOE, "Polymers and Organometallic Materials Research," 1/1/91 - 12/31/91, \$122,528

Stupp - NSF, "Ploymeric Liquid Crystal Research," 1/1/91 - 12/31/91, \$103,193

Stupp - Air Force, "Polymers and Organometallic Materials Research," 4/1/90 - 4/1/92, \$73,559

<u>Tucker</u> - Gen Corp Research Division, "Simulation of Compression Molding," 1/90- 12/91, \$40,000.

## OTHER SPONSORED RESEARCH (continued)

<u>Tucker</u> - Sundstrand Aviation, "Residual Stresses in Composite Parts," 6/90 - 6/91, \$40,000.

<u>Tucker</u> - Cray Valley S.A., "Simulation of Resin Transfer Molding," 8/90 - 8/91, \$40,000.

<u>Tucker</u> - Exxon Chemical, "Structural Thermoplastic Composites," 6/90 - 6/91, \$55,000.

<u>Tucker</u> - Dow Chemical Co., "Fluid Micromechanics of Resin Transfer Molding," 6/91 - 6/92, \$50,000.

White - Alliant Techsystems, "Hybrid Aluminum/Graphite/Epoxy Composite Components," 6/1/91 - 5/31/92, \$37,500.

White - Army Corp. of Engineers, "Smart Composite Materials for Construction Applications," 6/1/91 - 1/31/92, \$20,000.

#### LIST OF TECHNICAL REPORTS AND PUBLICATIONS

- 1. JOURNAL PUBLICATIONS 2. BOOKS
- 3. TECHNICAL REPORTS 4. PRESENTATIONS
- 1. Papers refereed and published in professional journals
- A. W.-L. Chen, A. Miyase, S. S. Wang, and P.H. Geil, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature, Part I: Neat Resin Characterization," <u>Journal of Composite Materials</u> (submitted).
- A. W.-L. Chen, A. Miyase, S. S. Wang, and P.H. Geil, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature, Part II: Time-Temperature Dependent Matrix Behavior," <u>Journal of Composite Materials</u> (submitted).
- A. W.-L. Chen, A. Miyase, S. S. Wang, and P.H. Geil, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature, Part III: Structure and Thermomechanical Properties of AS4/J1 Polymer Composite," <u>Journal of Composite Materials</u> (submitted).
- S.H. Doong, J.E. Faoro, and D.F. Socie, "In-Plane Biaxial Compressive Deformation and Failure of E-Glass/Epoxy Laminates," <u>ASTM STP 1120</u>, pp.87-102 (1992).
- D. Dean, A. Miyase, and P.H. Geil, "The Interaction of Creep and Physical Aging in a Semicrystalline Thermoplastic Matrix Composite Above Tg," <u>Journal of Thermoplastic Matrix Composites</u> (submitted).
- R.J. Gaylord, T.E. Twardowski, and J.F. Douglas, (P.H. Geil) "The Localization Model of Rubber Elasticity and the Deformation of a Network Formed by Cross-Linking a Strained Melt," <u>Polymer Bulletin</u>, 20, 305 (1988).
- R. Hajali, D.A. Pecknold, and A. Agah-Tehrani, "Effect of Interface Cracks on Elastic-Plastic Response of BLAP Composites under Longitudinal Shear," <u>Composites Engineering</u> (submitted).
- L.N. Hjellming and J.S. Walker, "Thermal Curing Cycles for Composite Cylinders with Thick Walls and Thermoset Resins," <u>Journal of Composite Materials</u>, Vol. 23, pp.1048-1064 (1989).
- L.N. Hjellming and J.S. Walker, "Motion of Continuous Fibers Through a Newtonian Resinfor High Fiber Volume Fraction," <u>Journal of Composite Materials</u>, Vol. 24, pp.853-878 (1990).

### 1. Journal publications (continued)

- H.T. Hu, "Effect of In-plane Shear Nonlinearity on Buckling and Postbuckling Responses of Composite Laminate Plates and Shells," <u>Journal of Applied Mechanics</u>, ASME (submitted).
- H.T. Hu, "Influence of Shell Geometry on Buckling Optimization of Fiber-Composite Laminate Shells," <u>Journal of Pressure Vessel Technology</u>, Vol. 113, No. 3, pp.465-470, August (1991).
- C. Jones, "The Use of Surface Signal Enhancement Techniques in X-Ray Photoelectronic Studies of Carbon Fibre Surfaces," <u>Carbon</u>, <u>27</u>, 487 (1989).
- C. Jones and E. Sammann, "The Effect of Low Power Plasmas on Carbon Fibre Surfaces," Carbon, 28 (1990).
- C. Jones and E. Sammann, "The Effect of Low Power Plasmas on Carbon Fibre Surfaces; Part II: A Comparison Between Low and High Modulus Pan Based Fibres with Pitch Based Carbon Fibres," <u>Carbon</u>, <u>28</u> (1990).
- L.S. Li and P.H. Geil, "On the Morphology and Structure of J-1 Polymer, Poly [(bis-4,4'-dicyclohexyl-methane) n-dodecanediamide] Spherulites, <u>Polymer</u>, 32, 374 (1991).
- D. Lin and P.H. Geil, "The Effect of Thermal Treatment on the J1 Polymer and it's Composite," <u>Journal of Thermoplastic Matrix Composites</u>, in press (1991).
- G. Liu and J.K. Shang, "Cyclic Strengthening of Carbon Fiber Reinforced Polymer Matrix Composites," to be published in <u>ASTM/STP</u>, November (1992).
- A. Miyase and S.S. Wang, "Interlaminar Creep of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperature," (in preparation).
- T.J. Moon and J.S. Walker, "Effects on Permeability of Fiber Clustering Induced by Resin Flow," <u>Journal of Composite Materials</u>, submitted for publication (1992).
- Y. Nakajo, "Creep Buckling of Thermoplastic-Matrix Fiber Composites under Multiaxial Loading," <u>Proceedings of the 4th US-Japan Conference on Composites Materials</u>, (1986).
- L.J. Neergaard, J. Homeny, and K. Karasek, J.T. Donner and S.A. Bradley, "Characterization of b-Si<sub>3</sub>N<sub>4</sub> Whiskers," <u>Journal of American Ceramic Society</u> (1988).
- M.E. Nichols, S.S. Wang, and P.H. Geil, "Creep and Physical Aging in a Polyimide Carbon Fiber Composites," <u>Journal of Macromolecular Science</u> (Physics), <u>B29</u>, 303 (1990).
- S. Pangrle, C.S. Wu, and P.H. Geil, "Low Temperature Relaxation of DGEBA Epoxy Resin: A TSDC Study," #138, in <u>Polymer Composites</u>, 10, 173 (1988).

### 1. Journal publications (continued)

- I.D. Parsons and B. Moradi, "Dimensional Analysis of Stiffened Composite Shells," <u>ASCE Journal of Engineering Mechanics</u>, February (1991).
- I.D. Parsons and B. Moradi, "Computing the Buckling Loads of Stiffened Shells," Computers and Structures (submitted).
- D.A. Pecknold, "3D Lamination Theory for Thick-Section Composites," <u>ASCE. Journal of Engineering Mechanics</u> (submitted).
- D.A. Pecknold and S. Rahmau, "A Simple Micromechanical Model for Fibrous Composites," <u>ASME Journal of Applied Mechanics</u> (submitted).
- R.T. Shield, "Variational Principles for Some Nonstandard Elastic Problems," <u>ASME J. of Applied Mechanics</u>, 54 (1987).
- N.R. Sottos, "Detecting Interphases Using Micro-Interferometric Measurements," <u>Experiments in Micromechanics of Failure Resistant Materials</u>, ed. by K.-S. Kim, AMD Vol. 130, pp.31-36 (1991).
- N.R. Sottos, "The Influence of the Fiber/Matrix Interface on Local Glass Transition Temperature," <u>Polymer Blends. Solutions and Interfaces</u>, ed. by I. Noda, Elsevier, (in press).
- R.J. Stango and S.S. Wang, "Representation of Viscoelastic Constitutive Properties of Fiber Composite Laminates," <u>Journal of Composite Science and Engineering</u> (1988).
- S.I. Stupp and S. Bhama, "Liquid Crystal Polymer-Carbon Fiber Composites. Molecular Orientation," Polymer Engineering Science, 30. (4), 228 (1990).
- S.I. Stupp and S. Bhama, "Grafting of a Liquid Crystal Polymer on Carbon Fibers," Polymer Engineering and Science Journal, 30, (10), 603 (1990).
- S.I. Stupp and S. Bhama, "Synthesis of Functionalized Side Chain Liquid Crystal Polymer," <u>Macromolecules</u>. (submitted).
- S.I. Stupp and S. Bhama, "Liquid Crystal Polymers with Functionalized Side Chains: Phase Transitions and Ordering on Fiber Surfaces," <u>Macromolecules</u> (submitted).
- T.E. Twardowski and R.J. Gaylord, (P.H. Geil) "The Localization Model of Rubber Elasticity and the Stress-Strain Behavior of a Network Formed by Cross-Linking a Deformed Melt II. Equibiaxial Extension and Pure Shear," <u>Polymer Bulletin</u>, 21, 393 (1989).
- T.E. Twardowski and P.H. Geil, "A Highly Fluorinated Epoxy Resin I: Post-Curing and Transition Behavior," <u>Journal of Applied Polymer Science</u>, 41, 1047 (1990).
- T.E. Twardowski and P.H. Geil, "A Highly Fluorinated Epoxy Resin II: Behavior in Blend Applications, Journal of Applied Polymer Science, 42, 69 (1991).

### 1. Journal publications (continued)

- T.E. Twardowski and P.H. Geil, "A Highly Fluorinated Epoxy Resin III: Behavior in Blend Applications, <u>Journal of Applied Polymer Science</u>. 42, 1721 (1991).
- T.E. Twardowski and P.H. Geil, "Curing in Thick Section Composite Laminates, Experiment and Simulation, <u>Journal of Composite Materials</u>, (submitted).
- T.E. Twardowski and O. Kramer, (P.H. Geil) "Elastic Contributions from Chain Entangling and Chemical Cross-links in Elastomer Networks in the Small Strain Limit," <u>Macromolecules</u>, 24, 5769 (1991).
- S.S. Wang and C.Y. Wang, "Multi-layered 3-D Hybrid Finite Element Analysis of Composite Plates and Shells," presented at the <u>24th AlAA/ASME/ASCE AHS Structures</u>. <u>Structural Dynamics and Materials Conference</u>, April (1988).
- S.S. Wang and F.G. Yuan, "An Analysis of Delamination in Fiber Composite Laminates Under Bending," Presented at <u>24th AIAA/ASME/ASCE AHS Structures, Structural Dynamics and Materials</u>.
- S.S. Wang, "Deformation and Failure of Thermoplastic Matrix Composites at Elevated Temperatures," Polymer Materials Science and Engineering, 59.
- S.S. Wang and P. Desoutter, "Elevated-Temperature Crack Growth and Fracture in Thermoplastic Polymers," <u>International Journal of Fracture</u> (in press).
- S.S. Wang and P. Desoutter, "Transverse Crack Growth and Fracture of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures," <u>Journal of Composite</u> <u>Materials</u> (in press).
- Z.Q. Jerry Wang and D.F. Socie, "Failure Strength and Damage Mechanisms of E-Glass/Epoxy Laminates under In-Plane Biaxial Compressive Deformation," Journal of Composite Materials, submitted (1991).

### 2. Books (and sections thereof) published

- P.H. Geil, A. Miyase, and S.S. Wang, "High Temperature Deformation and Fracture of Thermoplastic Composite Material," Marcel Dekker Inc., New York (in preparation).
- H.T. Hu, "Buckling Optimization of Fiber-Composite Laminate Shells with Large Deformation," <u>Optimization of Structural Systems and Industrial Applications</u>, Edited by Hernandez, S., and Brebbia, C. A., Co-published by Computational Mechanics Publications, Southampton and Elsevier Science Publishers Ltd, N.Y., pp.555-566 (1991).
- H.T. Hu and S.S. Wang, "Effect of In-plane Shear Nonlinearity on Buckling Optimization of Fiber-Composite Laminate Shells," <u>Composites: Design. Manufacture. and Application</u>, Edited by Tsai, S. W. and Springer, G. S., Society for the Advancement of Materials and Process Engineering, Covina, California, pp.4A1-4A12 (1991).

### 3. Technical report, non-refereed papers

(also, see attached listing of the Center's technical reports)

- D. Dean, A. Miyase, and P.H. Geil, "The Interaction of Creep and Physical Aging in a Semicrystalline Thermoplastic Matrix Composite Above Tg," <u>Proceedings American Society of Composites VI</u>, Technomic Publishing Co., pp.370 (1991).
- D. Hiemstra and N.R. Sottos, "Thermally Induced Micro-Cracking in Polymer Matrix Composites," <u>Proceedings of the 22nd Midwestern Mechanics Conference</u>, Rolla, MO, October (1991).
- V. Le Bonheur (MS Thesis) S. Stupp, "Molecular Engineering of Carbon Fiber/Epoxy Interphases: Adsorption, Grafting and Transfer of Langmuir-Blodgett Films of Liquid Crystalline Monomer and Its Polymer on Carbon Fibers," Univ. of Illinois (1991).
- D. Lin and P.H. Geil, "The Effect of Thermal Treatment on the J1 Polymer and it's Composite," <u>Proceedings American Society of Composites VI</u>, Technomic Publishing Co., pp.154 (1991).
- D. Lin, D. Dean, and P.H. Geil, "Regular, Adjacent Reentry Folding in Single Crystals of a Liquid Crystal Polymer," (in preparation).
- I.D. Parsons with B. Moradi, "Modeling Considerations for the Buckling of Stiffened Shells," <u>Proceedings of the CSME Mechanical Engineering Forum 1990</u>, vol. 2, pp.211-215 (1990).
- I.D. Parsons with B. Moradi, "A Comparison of Modeling Techniques for the Buckling of Stiffened Shells," <u>Proceedings of the ASCE Engineering Mechanics Speciality Conference</u>, May (1991).
- I.D. Parsons with B. Moradi, "The Influence of Material Anisotropy on the Buckling of Composite Structures, "Proceedings of the NSFU.S.-Korea-Japan Seminar on Frontier R&D for Constructed Facilities," Honolulu, Hawaii, October (1991).
- I.D. Parsons, "The Effect of Material Orthotropy on the Buckling and Postbuckling Behavior of Cylindrical Shells," <u>Recent Advances in Structural Mechanics</u>, PVP-vol. 225/NE-vol. 7, pp.27-32, ASME, December (1991).
- D.A. Pecknold and S. Rahman, "Nonlinear Compression Response of Thick-Section Laminates Using Micromechanics and Homogenization," <u>ASTM Symposium on Compression Response of Composite Structures</u>, Miami, Florida, Nov. (1992).
- N.R. Sottos, "Detecting Interphases Using Micro-Interferometric Measurements," <u>Proceedings of the ASME Winter Annual Meeting</u>, Atlanta, GA., Nov. (1991).
- N.R. Sottos, "The Influence of the Fiber/Matrix Interface on Local Glass Transition Temperature," <u>Proceedings of the VERP Symposium on Polymer Solutions</u>, Blends and <u>Interfaces</u>, Elsevier (1991).

- T.E. Twardowski and P.H. Geil, "Curing in Thick Section Composite Laminates, Experiment and Simulation," <u>Proceedings American Society of Composites VI</u>, Technomic Publishing Co., pp.13 (1991).
- S.S. Wang, E.C. Klang, and A. Miyase, "Viscoelastic Shear of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures" (in preparation).

#### 4. Presentations

#### **Annual Program Review Meetings:**

- 1st ONR URI review meeting, November 12, 1986, University of Illinois.
- 2nd ONR URI review meeting, October 17-19, 1988, University of Illinois.
- 3rd ONR URI review meeting, February 5-7, 1990, University of Illinois.
- Mini- review meeting, June 11, 1991, (Barsoum, Rajapakse) University of Illinois.
- 4th ONR URI review meeting, September 12-13, 1991
  David Taylor Research Center in Annapolis, Maryland.
- D. Dean and P.H. Geil, "Interaction of Physical Aging and Long Term Creep in Thermoplastic Matrix Composite above T<sub>g</sub>," <u>American Physics Society</u>, March 1991 and <u>American Composite Society</u>, Oct. (1991).
- H.T. Hu, "Influence of Shell Geometry on Buckling Optimization of Fiber-Composite Laminate Shells," <u>32nd AIAA/ASME/ASCE/AHS/ASC Structures</u>. Structural Dynamics and Materials Conference, Baltimore, Maryland, April 8-10 (1991).
- H.T. Hu, "Buckling Optimization of Fiber-Composite Laminate Shells with Large Deformation," <u>Second International Conference on Computer Aided Optimum Design of Structures</u>, Cambridge, Massachusetts, June 25-27 (1991).
- H.T. Hu and S.S. Wang, "Effect of In-plane Shear Nonlinearity on Buckling Optimization of Fiber-Composite Laminate Shells," <u>Eighth International Conference on Composite</u> <u>Materials</u>, Honolulu, Hawaii, July 15-19 (1991).
- H.T. Hu, "Effect of In-plane Shear Nonlinearity on Buckling and Postbuckling Responses of Composite Laminate Plates and Shells," will be presented in <u>Symposium on Enhancing Analysis Techniques for Composite Materials</u>. ASME Winter Annual Meeting, Atlanta, Georgia, December 1-6 (1991).
- D. Lin and P.H. Geil, "Structure-Property-Thermal History Relationship in J1 Polymer and AS4/J1," Composites, APS, March 1991 and ACS, Oct. (1991).

### 4. Presentations (continued)

- G. Liu and J.K. Shang, "Cyclic Strengthening of Carbon Fiber Reinforced Polymer Matrix Composites," to be presented in the <u>ASTM Symposium on Compression Response of Composite Structures</u>, Nov. 16-17, Miami, FL. (1992).
- B. Moradi and I.D. Parsons, "The Influence of Material Anisotropy on the Buckling of C C Structures," <u>U.S.-Korea-Japan Seminar on Frontier R&D for Constructed Facilities</u>, Honolulu, Hawaii, October (1991).
- Z.Q. Jerry Wang and D.F. Socie, "Biaxial Testing of G-10 Composite Laminates," ASTM 11th Symposium on Composite Testing, May 4-5, 1992, Pittsburgh, PA.
- Z.Q. Jerry Wang and D.F. Socie, "Biaxial Compression Testing of a Woven Composite Laminate," Japan-US Conference on Composite Materials, June 22-25, 1992, Orlando, FL.
- Z.Q. Jerry Wang and D.F. Socie, "Biaxial Compression Behavior of G-10 Laminates with Cutouts," ASTM Symposium Compressive Response of Composite Structures, Nov. 16-17, 1992, Miami, FL.
- M.E. Nichols and P.H. Geil, "Creep and Physical Aging in a Polyamideimide-Carbon Fiber Composite, <u>Journal of Macromolecular Science</u>, <u>B29</u>. 303, American Physics Society (1989).
- I.D. Parsons, "The Buckling of Stiffened Composite Shells," <u>11th U.S. National Congress of Applied Mechanics</u>, Tucson, AZ, May (1990).
- I.D. Parsons, "Modeling Considerations for the Buckling of Stiffened Shells," <u>CSME Mechanical Engineering Forum</u>, Toronto, Canada, June (1990).
- I.D. Parsons, "The Buckling Behavior of Stiffened Composite Shells," <u>Korea Advanced Institute of Science and Technology</u>, Seoul, Korea, December (1991).
- I.D. Parsons, "A Comparison of Modeling Techniques for the Buckling of Stiffened Shells," <u>ASCE Engineering Mechanics Speciality Conference</u>, Columbus, OH, May (1991).
- I.D. Parsons, "The Effect of Material Orthotropy on the Buckling and Postbuckling Behavior of Cylindrical Shells," 1991 ASME WAM, Atlanta, GA. December (1991).
- D.A. Pecknold, "3D Nonlinear Modeling of Thick-Section Composites," DTRC Carderock/Annapolis, Nov. 27/28 (1990).
- D.A. Pecknold, A. Agah-Tehrani, and R. Hajali, "Effect of Interface Cracks on Elastic-Plastic Response of B/AL Composites under Longitudinal Shearing," <u>International Congress of Theoretical and Applied Mechanics</u>, Haifa, Israel, August (1992).
- D.A. Pecknold and R. Hajali, "Combined Micromechanical and Structural Synthesis Modeling for Multi-Layered Composite Structures," <u>International Congress of Theoretical and Applied Mechanics</u>, Haifa, Israel, August (1992).

### 4. Presentations (continued)

- N.R. Sottos, "The Influence of the Fiber/Matrix Interface on Local Glass Transition Temperature, <u>The Procter and Gamble UERP Symposium on Polymer Solutions, Blends and Interfaces, American Chemical Society Meeting</u>, New York, September (1991).
- N.R. Sottos, "Detecting and Tailoring the Interphase in Polymer Matrix Composites," ASME WA Meeting. Symposium on Experiments in Micromechanics of Failure Resistant Materials, Atlanta, GA, December (1991.).
- T.E. Twardowski and P.H. Geil, "Curing in Composite Laminates: Thick Sections and Simulations," <u>ACS</u>, Oct. (1991).
- S. White, "Thermo-Chemical Modeling of an Economic Manufacturing Technique for Composite Structures," <u>Proceedings of the 37th SAMPE Symposium and Exhibition</u>, Anaheim, CA, March 9-12 (1992).

### **Technical Papers and Reports**

#### Report #

#### Author(s) and Titles

- 89-01 A.W.-L. Chen, A. Miyase, P.H. Geil and S.S. Wang, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature; Part I: Neat Resin Structure Characterization."
- 2) 89-02 A.W.-L. Chen, A. Miyase, P.H. Geil and S.S. Wang, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature; Part II: Time-Temperature Dependent Matrix Behavior."
- 3) 89-03 A. Miyase, S.S. Wang, A.W.-L Chen and P.H. Geil, "Anelastic Deformation of a Thermoplastic-Matrix Fiber Composite at Elevated Temperature; Part III: Structure and Thermomechanical Properties of AS4/J1 Composite."
- 4) 89-04 S. Bhama and S.I. Stupp, "Liquid Crystal Polymer-Carbon Fiber Composites. Molecular Orientation," Polymer Engineering and Science, 30, (4), 228 (1990).
- 5) 89-05 S. Pangrle, C.S. Wu and P.H. Geil, "Low Temperature Relaxations of DGEBA Epoxy Resins: A TSDC Study," <u>Polymer Composites</u> (in press).
- 6) 89-06 C. Jones, "The Use of Surface Signal Enhancement Techniques in X-Ray Photoelectronic Studies of Carbon Fibre Surfaces," <u>Carbon</u>, 27, 487 (1989).
- 7) 89-07 J. Homeny and L.J. Neergaard, K.R. Karasek, J.T. Donner and S.A. Bradley, "Characterization of b-Si<sub>3</sub>N<sub>4</sub> Whiskers," <u>Journal of American Ceramic Society</u>, accepted (1989).
- 8) 89-08 L.J. Neergaard and J. Homeny, "Mechanical Properties of Beta-Silicon Nitride Whisker/Silicon Nitride Matrix Composites," <u>Ceramic Engineering Science Proceedings</u>, accepted (1989).
- 9) 89-09 R.J. Gaylard, T.E. Twardowski and J.F. Douglas, "The Localization Model of Rubber Elasticity and the Deformation of a Network Formed by Cross-Linking a Strained Melt," Polymer Bulletin, 20, 305 (1988).
- 10) 89-10 T.E. Twardowski and R.J. Gaylord, "The Localization Model of Rubber Elasticity and the Stress-Strain Behavior of a Network Formed by Cross-Linking a Deformed Melt II. Equibiaxial Extension and Pure Shear," Polymer Bulletin, 21, 393 (1989).
- 11) 89-11 L.N. Hjellming and J.S. Walker, "Thermal Curing Cycles for Composite Cylinders with Thick Walls and Thermoset Resins," <u>Journal of Composite Materials</u>, in press (1989).
- 12) 89-12 L.N. Hjellming and J.S. Walker, "Motion of Continuous Fibers Through a Newtonian Resin for High Fiber Volume Fraction," <u>Journal of Composite Materials</u>, submitted (1989).
- 13) 89-13 T.E. Twardowski (thesis) and P.H. Geil, "Characterization Studies of Fluorinated Epoxy Resins: Naval Experimental Resin C8/1SA as a Structural Material and for Use in Blends and Composites."

#### Technical Reports (continued)

#### Report #

#### Author(s) and Titles

- 14) 89-14 R.T. Shield, "Variational Principle for some Nonstandard Elastic Problems," <u>ASME Journal of Applied Mechanics</u>, Vol. <u>54</u>, pp. 768-771 (1987).
- 15) 89-15 A. Miyase and S.S. Wang, "Transverse Creep of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures" (in preparation).
- 16) 89-16 C. Jones and E. Sammann, "The Effect of Low Power Plasmas on Carbon Fibre Surfaces," Carbon, 28 (1990).
- 17) 89-17 C. Jones and E. Sammann, "The Effect of Low Power Plasmas on Carbon Fibre Surfaces; Part II: A Comparison Between Low and High Modulus Pan Based Fibres with Pitch Based Carbon Fibres," <u>Carbon</u>, 28 (1990).
- 18) 89-18 C. Jones and E. Sammann, "The Effect of Low Power Plasmas on Carbon Fibre Surfaces."
- 19) 89-19 S.S. Wang and P. Desoutter, "Elevated-Temperature Crack Growth and Fracture in Thermoplastic Polymers," <u>International Journal of Fracture</u> (in press).
- 20) 89-20 S.S. Wang and P. Desoutter, "Transverse Crack Growth and Fracture of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures," Journal of Composite Materials (in press).
- 21) 89-21 Y.S. Yuan and S.S. Wang, "An Estimation of Temperature-Dependent Transversely Isotropic Thermoelastic Properties of Single-Crystal SiC Whiskers."
- 22) 89-22 Y.S. Yuan and S.S. Wang, "High-Temperature Thermoelastic Constitutive Theories for Random Whisker-Reinforced Ceramic Composites, Part I: Under Small Temperature Change."
- 23) 89-23 Y.S. Yuan and S.S. Wang, "High-Temperature Thermoelastic Constitutive Theories for Random Whisker-Reinforced Ceramic Composites, Part II: Under Large Temperature Change."
- 24) 89-24 Y. Nakajo and S.S. Wang, "Creep Buckling of Thermoplastic-Matrix Fiber Composites Under Biaxial Loading," <u>Journal of Thermoplastic Composite Materials</u>, Vol. 2, No. 3 pp.172-215 (1989)
- 25) 89-25 H-T. Hu and S.S. Wang, "Optimization for Buckling Resistance of Fiber-Composite Laminate Shells With and Without Cutouts," <u>AIAA Journal</u> (1990).
- 26) 89-26 S.S. Wang, J. Blondet, A. Miyase, and K.B. Su, "Interlaminar Fatigue Crack Growth in a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures."
- 27) 89-27 A. Agah-Tehrani and S. Wu, "The Influence of Anisotropy of the Microstructure on the Overall Elastic-Plastic Response of Fibrous Metal-Matrix Composites."
- 28) 89-28 A. Agah-Tehrani and S. Gunawardena, "Micro- and Macrobifurcation of Fibrous Metal-Matrix Composites Under Uniaxial Compression."

#### Technical Reports (continued)

#### Report #

#### Author(s) and Titles

- 29) 89-29 C.D. Balis and S.S. Wang, "Micromechanics Analysis of Fiber Toughening in Ceramic-Matrix Composites."
- 30) 89-30 S.S. Wang, E.C. Klang, and A. Miyase, "Viscoelastic Shear of a Thermoplastic-Matrix Fiber Composite at Room and Elevated Temperatures" (in preparation).
- 31) 89-31 S. Bhama and Samuel I. Stupp, "Grafting of a Liquid Crystal Polymer on Carbon Fibers," Polymer Engineering and Science, 30, (10), 603 (1990).
- 32) 90-01 T.-P. Yu and S.S. Wang, "Analysis of Cracks in Nonlinear Fiber Composite Materials."
- 33) 90-02 L. Rouxel and S.S. Wang, "Buckling and Postbuckling Analyses of Fiber-Composite Laminate Plates Under Biaxial Compression."
- 34) 90-03 M.E. Nichols, S.S. Wang and P.H. Geil, "Creep and Physical Aging in a Polyamideimide-Carbon Fiber Composite."
- 35) 90-04 Christopher Mangin, "R-Curve Behavior for Silicon Carbide Whisker Reinforced Aluminum Oxide Composites."
- 36) 90-05 Satya Bhama Sastri (PhD Thesis) and S.I. Stupp, "Novel Interphases: Synthesis, Molecular Orientation and Grafting of Liquid Crystal Polymers on Carbon Fibers" Univ. of Illinois (1990).
- 37) 90-06 S.H. Doong, J.E. Faoro, D.F. Socie and S.S. Wang, "Development of a Test System and Experimental Method for In-Plane Biaxial Compressive Deformation and Failure of Fiber Composite Materials."
- 38) 90-07 M. Gosz, B. Moran and J.D. Achenbach, "Effect of a Viscoelastic Interface on the Transverse Behavior of Fiber-Reinforced Composites."
- 39) 90-08 M. Buckley and J. Homeny, "Tensile/Compressive Fatigue and Interface Characterization of A1<sub>2</sub>0<sub>3</sub> Fiber/A1-2.5% Li Alloy Metal Matrix Composite."
- 40) 90-09 Kiet Van Chau, "Static and Cyclic Compressive Fatigue of Thermoplastic-Matrix Composite in Ambient and Moisture Environments."
- 41) 90-10 Rami Hajali and Su Su Wang, "Nonlinear Behavior of Fiber Composite Materials and Its Effect on the Postbuckling Response of Laminated Plates."
- 42) 90-11 B. Moradi and I.D. Parsons, "A Study of the Computation of the Buckling Behavior of Some Ring Stiffened Shells."
- 43) 90-12 B. Moradi and I.D. Parsons, "Dimensional Analysis of the Buckling of Stiffened Composite Shells."
- 44) 91-01 Hong Teng and A. Agah-Tehrani, "Interfacial Slippage of a Unidirectional Fiber Composite Under Longitudinal Shearing."

#### Technical Reports (continued)

#### Report #

#### Author(s) and Titles

- 45) 91-02 A. Agah-Tehrani and Hong Teng, "A Model for Wet Filament Winding of Composite Cylinders with Arbitrary Thickness."
- 46) 91-03 Amanda Spindel and P. Geil, "Effects of Physical Aging on Stress Relaxation and Fracture Toughness of a Polymer and its Carbon Fiber Composite."
- 47) 91-04 Hsuan-Teh Hu, "Influence of Shell Geometry on Buckling Optimization of Fiber-Composite Laminate Shells."
- 48) 91-05 Hsuan-Teh Hu, "Influence of In-Plane Shear Nonlinearity on Buckling and Postbuckling Responses of Composite Plates and Shells."
- 49) 91-06 David Lin, "Characterization of Thermoplastic J1 Polymer and its Graphite Fiber AS4/J1 Matrix Composites."
- 50) 91-07 D.R. Dean, A. Miyase, P.H. Geil, "The Interaction of Physical Aging in a Semicrystalline Thermoplastic Matrix Composite above Tg," presented at the American Society of Composites meeting, Albany, New York, Oct. (1991).
- 51) 91-08 T.E. Twardowski, S.E. Lin, P.H. Geil, "Curing in Composite Laminates: Thick Sections and Simulations."
- 52) 91-09 Z. Zhang, A. Agah-Tehrani, "Transient Thermal Stresses During Cooling of Cross-Ply Thermoset Composite Cylinders."
- 53) 91-10 T.E. Twardowski and P.H. Geil, "Some Molecular Contributions and Curing Considerations: Physical Properties of Thermoset Network Materials," Ph.D. Thesis, University of Illinois, February (1992).
- 54) 91-11 T.E. Twardowski and P.H. Geil, "The Effect of Chain Stiffness in Lightly Cross-Linked Epoxies," submitted to Journal of Macromolecular Science - Pure and Applied Chemistry, February (1992).

# Student Degree List

	STUDENT	ADVISOR	Masters	PhD
1	Aggour, Hany	Wang, S.S.		
2	Althauser, John	Stubbins		May-92
3	Bhama, Satya	Stupp		May-90
4	Burkhardt, John	Wang, S.S.		
5	Dean, Derrick	Geil	May-92	
6	DeMoss Timothy	Sottos	Sep-91	
7	Deppish, Carl	Economy	Aug-92	
8	Duban, James	Miyase	_	
9	Folmer, David	Socie	May-92	
10	Gillette, Rosemary	Walker	·	
11	Hjellming, Liza	Walker		
12	Huang, Ching-Hua	Homeny	May-91	
13	Jun, Al∈ ander	Corten	-	
14	Jur, Joseph	Sehitoglu	Dec-91	
15	Karayaka, Metin	Sehitoglu		Oct-91
16	Kelly, Mark	Homeny		
17	Kim, Yeong	White		Dec-94
18	Kocher, Chris	Agah-Tehrani		
19	Lebonheur, Vas.	Stupp	Aug-91	
20	Li, Li	Sottos	•	
21	Lim, Yeow B.	Parsons		May-92
22	Lin, David	Geil		Aug-91
23	Liu, Gang	Shang		Dec-94
24	Moradi, Behrooz	Parsons		May-92
25	Neergaard, Lynn	Homeny		
26	Nelson, Daniel	Zak	Dec-87	
27	Nichols, Mark	Geil	Aug-89	
28	Pollock, Gerry	Zak	Aug-89	
29	Rahman, Shahzad	Pecknold	•	Dec-91
30	Rouxel, Laurent	Wang		Aug-90
31	Song, Felix	Wang		•
32	Spindel, Amand	Geil	May-91	
33	Talmage, Gita	Walker	•	
34	Teng, Hong	Tucker		May-93
35	Thiel, George	Shield		•
36	Twardowski, T	Geil	Oct-88	Dec-91
37	Wang, Zhiqi	Socie		Aug-94
38	Wu, San-Jarn	Wang		J
39	Yogeswaren, Elan	Wang		Dec-91
40	Zhang, Zhibin	White		May-94
41	Zhengxian, Bai	Wang		•
	Total Projected Degre		12	15

# FACULTY / POSTDOCTORAL DATA

	Postdoctoral	Title
1	Bouadi, Hacene	Postdoc Asst
2	Doong, Shiing-Hwa	Postdoc Asst
3	Jansson, Stefan	Postdoc Asst
4	Mikata, Yozo	Postdoc Asst
5	Moon, Tessie	Postdoc Asst
6	Shibuya, Yotsugi	Postdoc Asst
7	Wang, C Y	Postdoc Asst
8	Yu, Tung-Pei	Postdoc Asst
9	Yuan, Fuh-Gwo	Postdoc Asst
	Faculty	Title
1	Agah-Tehrani, Abdi	Asst Prof
2	Corten, Herbert	Prof
3	Economy, James	Prof
4	Geil, Phil	Prof
5	Homeny, Joseph	Asst Prof
6	Hu, Hsuan-Teh	Res Engineer
7	Jones, Carol	Sr Res Engr
8	Kurath, Peter	Sr Res Engr
9	Miyase, Aki	Prin Res Eng
10	Parsons, Dennis	Asst Prof
11	Pecknold, David	Prof
12	Sehitoglu, Huseyin	Assoc Prof
13	Shang, Jian-Ku	Asst Prof
14	Shield	Prof
15	Socie, Darrell	Prof
16	Sottos, Nancy	Asst Prof
17	Stubbins, James	Assoc Prof
18	Stupp, Samuel	Prof
19	Tucker, Charles	Prof
20	Walker, John	Prof
21	Wang, S S	Prof
22	White, Scott	Asst Prof
23	Zak, Adam	Prof

# Subcontractor Final Technical Report Executive Summaries of Final Technical Reports

	University	Project Director/s
1.	Drexel University	Michael J. Koczak Ihab Kamel
2.	Iowa State University	Donald O. Thompson R. Bruce Thompson
3.	University of Maryland	Sung W. Lee
4.	Michigan State University	Martin C. Hawley Lawrence T. Drzal Jes Asmussen, Jr. James V. Beck
5.	Northwestern University	Jan D. Achenbach B. Moran
6.	University of Pennsylvania	Zvi Hashin
7.	Virginia Polytechnic Institute	Daniel Post

# UNIVERSITY RESEARCH INITIATIVE PROGRAM, COMPOSITE MATERIALS

# HIGH PERFORMANCE COMPOSITE MATERIALS, PART I. RF PLASMA MODIFICATION OF GLASS FIBER POLYPHENYLENE SULFIDE INTERFACE

FINAL REPORT

**SUBMITTED** 

TO

UNIVERSITY OF ILLINOIS AT URBANA-CHAMPAIGN COLLEGE OF ENGINEERING 100-6 TALBOT LABORATORY 104 SOUTH WRIGHT STREET URBANA, ILLINOIS

BY

DR. MICHAEL KOCZAK AND DR. IHAB KAMEL DREXEL UNIVERSITY DEPARTMENT OF MATERIALS ENGINEERING PHILADELPHIA, PA 19104

**JANUARY 30, 1992** 

MICHAEL KOCZAK, PROFESSOR PRINCIPAL INVESTIGATOR

215-895-2327

IHAB KAMEL, PROFESSOR CO-PRINCIPAL INVESTIGATOR

215-895-2322

# TABLE OF CONTENTS

LIS	T OF	PUBLICATIONS	
1.	INT	RODUCTION	1
2.	BAG	CKGROUND	4
	2.2	Fiber Surface Treatments to Improve Interfacial Bonding	4
	2.3	Glow Discharge	6
	2.4	Surface Modifications Induced By Non-Polymer Forming Plasmas	6
3.	API	PROACH	12
4.	ARC	GON PLASMA TREATMENT	15
	4.1	Experimental Procedure	15
	4.2	Results and Discussion	16
	4.3	Summary	18
5.	PLA	SMA POLYMERIZATION	19
	5.1	Experimental Procedure	19
	5.2	Plasma Polymerization of Allylamine	20
	5.3	Plasma Polymerization of Hexamethyldisiloxane	25
	5.4	Summary	28
6.	MO	RPHOLOGY AND STABILITY OF THE COATINGS	30
	6.1	Experimental Procedure	30
6.2	Coa	ting Morphology	30
	6.3	Thermal Stability	31
	6.4	Environmental Stability	32
	6.5	Mechanical Stability	33
	6.6	Summary	33
7.	POS	T-GRAFTING ARGON PLASMA	33
	7.1	Experimental Procedure	33
	7.2	Results and Discussion	34
	7.3	Summary	35
8.	INT	ERFACE TESTING	
	8.1	Existing Techniques to Measure the Bond Strength at the Interface	
	8.2	Surface Coating Resistance: A New Technique to Test the Interface	
	8.3	Experimental Procedure	
	8.4	Results and Discussion	
	8.5	Summary	
	-	•	

# UNIVERSITY RESEARCH INITIATIVE PROGRAM COMPOSITE MATERIALS

HIGH PERFORMANCE MATERIALS

COMPOSITE MATERIALS

1st, 2nd & 3rd QUARTERLY REPORTS

BY

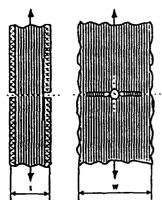
DREXEL UNIVERSITY

#### SUBMITTED TO

UNIVERSITY OF ILLINOIS at URBANA-CHAMPAIGN
COLLEGE OF ENGINEERING
100-6 TALBOT LABORATORY
104 SOUTH WRIGHT STREET
URBANA, ILLINOIS



DREXEL UNIVERSITY
COLLEGE OF ENGINEERING
PHILADELPHIA, PA 19104



M.J. Koczak, Professor and Principal Investigator Materials Engineering (215) 895-2329

Co-Principal Investigators:

J. Awerbuch, Department of Mechanical Engineering & Mechanics

I. Kamel, Department of Materials Engineering

A.Lau, Department of Mechanical Engineering & Mechanics



#### **EXECUTIVE SUMMARY**

Significant improvements in the strength and performance of advanced resin and metal matrix composites are founded on a thorough understanding of the critical aspects of pertinent composite mechanics and materials science coupled with establishment of the engineering requirements for optimum composite design to meet performance demands. The University Research Initiative in high performance composite materials incorporates the contribution expertise of the Materials Engineering and Mechanical Engineering and Mechanics Departments at Drexel University to the University of Illinois University Research Initiative program. Coupled with the David Taylor Naval Ship R & D Center at Annapolis, MD and the Naval Air Development Center at Warmininster, PA. Drexel University addresses those problem areas key to significantly improved hybrid thermoplastic and epoxy composites and composite structures at the macroscopic and microscopic levels. Specifically, lamina hybrid composites (LHC) and fiber hybrid composite (FHC) composite structures are considered. The configuration and packing of the fiber component play the dominant role in determining the properties of any composite. Specific attention has been directed to hybrid composite structures which will be examined both by micromechanical modelling and experimentally in terms of measured composite properties in sufficient detail to establish a base for optimizing larger composite structures. Hybrid composites, e.g. ARALL configurations may have significant benefit in damage tolerance, fatigue resistance and formability.

The key issues emphasized at Drexel University for evaluation are (i) effects of processing variables, (ii) interface/interphase thermodynamics, kinetics and mechanics, (iii) material and defect characterization utilizing nondestructive, i.e. acoustic emission, techniques with micromechanics and micro-mechanisms of deformation and failure supporting the areas of fiber and lamina hybrid composites. The faculty and staff members involved include Dr. Ihab Kamel (i.e. Interfacial Modification by Plasma Processing), Drs. Michael J. Koczak and Murali Vedula (i.e. Processing and Evaluation of Fiber Hybrid Composites and Lamina Hybrid Composites), Dr. Jonathan Awerbuch (i.e. Tracking Progression of Delamination in Model Composites Through Acoustic Emission During Quasi-Static and Fatigue Loading) and Dr. Alan Lau (i.e. Micromechanics of Lamina Hybrid Composites). The combined mechanics, materials and chemistry team provides for a coordinated interdisciplinary approach to these composites isssues. For each of the research efforts, the progress and activities of the Fall, Winter and Spring quarters are detailed in the following report.

In summary, the overall goal is the establishment of a strong mechanics and materials science base for the development of high performance composites by considering (i) effects of processing variables, (ii) interface/interphase processing kinetics and thermodynamics, (iii) non- destructive defect and materials characterization,

constitutive equations with support in the analysis of the micromechanics and micromechanisms of hybrid composite performance.

# NDE for Thick Section Composites

ONR Quarterly Report July 1 - September 30, 1991 Contract #N00014-86-K-0799

> Iowa State University Center for NDE Applied Science Complex II 1915 Scholl Road Ames, IA 50011

# **IOWA STATE UNIVERSITY**

# Table of Contents

	Pag	e Number
Γask 1:	Elastic Properties of Thick Composites Emmanuel P. Papadakis and Yu Min Tsai	1
Γask 2:	Defect Characterization	6
Γask 3:	Discrete Defect Characterization	. 9

## **IOWA STATE UNIVERSITY**

TASK 1: ELASTIC PROPERTIES OF THICK COMPOSITES

**REPORT PERIOD:** July 1 - September 30, 1991

PRINCIPAL INVESTIGATOR: Emmanuel P. Papadakis

**INVESTIGATOR:** Yu-Min Tsai

STUDENT: Thadd Patton (through April, 1991)

#### **OBJECTIVE:**

To measure and report on the elastic moduli of thick, graphite-epoxy, filament-wound composites.

The purpose of this work is to permit the calculation of allowed elastic wave modes and directions for nondestructive evaluation of thick composites. In addition, the anisotropic elastic moduli so obtained will contain important information about the microstructure of the material and the vibration characteristics of structures made from it. As the material has been found to be highly anisotropic, it is expected that ultrasonic interrogation to find flaws will be complicated. To calculate the wave propagation phenomena which may be useful or detrimental, it is necessary to measure the elastic moduli of the material.

#### APPROACH:

The approach is to investigate the possibility of using an array of static as well as dynamic elastic measurement methods to determine the elastic moduli of the materials in the symmetry and configuration of the composites. The parts of greatest interest are cylinders with the symmetry axes defined as 1 = axial, 2 = hoop, and 3 = radial. With the theoretical expressions for the measurable variables available, it will be possible to pick a feasible set of measurements from which to calculate the nine elastic moduli of the materials.

Having a complete array of theories is of importance from two points of view:

- (1) The contracting agency wishes to be able to determine the moduli by using uncut right circular cylinders as manufactured rather than having to cut special specimens for laboratory measurement. To date, the measurements have been made on specimens cut analogous to crystals of similar local symmetry. One full-size cylinder has been obtained to continue the work, and the theories are necessary for the interpretation of measurements made in this geometry.
- (2) Certain deformations may prove particularly advantageous for flaw detection. These can only be identified and fully exploited with a proper theoretical foundation.

The array of theories and techniques can be subdivided as follows to provide specific approaches:

- A. Ultrasonic Traveling Bulk Waves
  - 1. Normal Incidence
  - 2. Oblique Incidence
  - 3. Surface Excitation by Line Contact
  - Surface Excitation by Oblique Immersion
- B. Ultrasonic Traveling Surface Waves
- C. Ultrasonic Traveling Lamb Waves
- D. Body Vibrations
- E. Static Stress-Strain

Progress on these techniques will be reported in the next section. Both theory and experiment will be mentioned.

# The ONR-URI Research on Composites

Final Technical Report submitted to the National Center for Composite Materials Research the University of Illinois at Urbana-Champaign

February 1992

S.W. Lee

Department of Aerospace Engineering University of Maryland

#### UNIVERSITY OF MARYLAND

# **Summary**

This report covers the ONR-URI research carried out by the University of Maryland over a three-year period (9/15/86-9/14/89). As a member of the University Consortium led by the University of Illinois at Urbana-Champaign, the University of Maryland conducted basic research on 1) mechanics of composite structures and 2) synthesis of new matrix materials.

The research on mechanics of composite structures covered both analysis and testing of structural components with emphasis on thick section composite laminates. In particular, the focus of research effort is to investigate and understand prebuckling, buckling response and failure behavior of thick section composite structures including the effects of local imperfections such as ply wrinkles or ply delamination. Our emphasis in the three-year period was to develop 1) computational tools necessary for analytical investigation and 2) testing facilities necessary for experimental investigation of thick section composite structures. For this purpose, a three dimensional solid finite element technique was developed. This finite element model is tailored for composite shell structures and is capable of handling large as well as small deflection. Dr. Vizzini and his student in the Department of Aerospace Engineering carried out experimental investigation of notch sensitivity of thick section composite specimen under compressive loading.

The objective of the research on matrix material was to synthesize new monomers that will undergo controlled expansions, so that they can be added to an epoxy resin to form a matrix for carbon fiber-epoxy composite that has superior impact strength, increased tensile and shear strength. In this important area, Dr. William J. Bailey (deceased) and his group in the Department of Chemistry made a significant progress. A measure of this success is the large number of papers they published in the refereed journals.

The funding provided by the ONR-URI program was instrumental to the establishment of the Composites Research Laboratory at the University of Maryland. The program also allowed us to obtain the matching funds from the University to acquire additional equipment. Experimental facilities developed at Maryland include microprocessor controlled autoclave with 3 ft diameter and 4 ft length working section, 220,000 lbs MTS uniaxial testing machine and an X-ray machine for nondestructive inspection.

More detail description of the research effort at the University of Maryland is given in the following sections.

#### MICHIGAN STATE UNIVERSITY

J. Adhesion, 1989, Vol. 29, pp. 63-79
Reprints available directly from the publisher
Photocopying permitted by license only
© 1989 Gordon and Breach Science Publishers, Inc.
Printed in the United Kingdom

# Effects of Microwave Processing on Fiber-matrix Adhesion in Composites†

RAJ K. AGRAWAL and LAWRENCE T. DRZAL\*

Department of Chemical Engineering, and Composite Materials and Structures Center, Michigan State University, East Lansing, MI 48824-1326, U.S.A.

(Received July 21, 1988; in final fc n November 16, 1988)

Experiments have been done using a single mode (TE<sub>111</sub>, 2.45 GHz) cylindrial microwave cavity with single fiber composite specimens. After obtaining a cure cycle with microwaves to match that achieved with a conventional thermal cure cycle as measured by tensile tests, dynamic mechanical analysis and differential scanning calorimetry, quantitative measurements of interfacial shear strength and physical properties have been carried out and compared with the results from conventional thermally-cured systems. Under the conditions studied for single fiber specimens, the fiber-matrix interfacial shear strength decreases slightly in both glass-epoxy and aramid-epoxy cases as compared with thermally-cured specimens. Graphite fiber-epoxy adhesion, on the other hand, increases significantly in these single fiber studies in microwave processed specimens as indicated by an increase in the interfacial shear strength. The failure mode changes from interfacial (thermal curing) to matrix failure.

KEY WORDS Microwave curing; interface/interphase; adhesion; fiber reinforced composite; interfacial shear strength; single fiber testing.

#### 1. INTRODUCTION

The processing of high performance and thick composite materials requires the transfer of energy efficiently into the polymer mass. The introduction of high temperature matrix materials necessitates the use of very high temperature processing equipment for the production of these new-generation composite materials if traditional thermal processing techniques are used. Electromagnetic heating of polymer systems is an alternative to conventional thermal processing. Microwave curing of composites offers the potential for the development of an extremely fast and versatile method of composite processing. Control of energy input, duration and location provides an inherent flexibility not present in any other technique. Limitations in thermal processing due to poor heat conductivity of polymers and accelerated thermal degradation at elevated temperatures due to

\* To whom all correspondence should be addressed.

<sup>†</sup> Presented at the 35th Sagamore Army Materials Research Conference, Manchester, New Hampshire, U.S.A., June 26-30, 1988.

# MICHIGAN STATE UNIVERSITY

Fiber-Matrix Adhesion and Its Effect on Composite Mechanical Properties: I. Inplane and Interlaminar Shear Behavior of Graphite/Epoxy Composites

MADHU S. MADHUKAR AND LAWRENCE T. DRZAL Composite Materials and Structures Center

mposue maieriais ana structures Cer Michigan State University East Lansing, MI 48824

Reprinted from Journal of COMPOSITE MATERIALS, Vol. 25-August . 91

### NORTHWESTERN UNIVERSITY

### FINAL TECHNICAL REPORT

# MICRO-MECHANICAL MODELLING OF DEFORMATION AND FRACTURE IN FIBER-MATRIX INTERPHASES

by

J.D. Achenbach and B. Moran

Center for Quality Engineering and Failure Prevention Northwestern University Evanston, IL 60208

#### submitted to

National Center for Composite Materials Research
University of Illinois at Urbana-Champaign

Contract Period: April 1, 1989 - September 30, 1989

Contract No.: N00014-86-K-0799, Subcontract No. 89-122

Contact: J.D. Achenbach (708) 491-5527,

B. Moran (708) 491-8793, moran@nuacc.acns.nwu.edu

#### NORTHWESTERN UNIVERSITY

# 1. Description of Scientific Research Goals

The overall behavior of fiber-reinforced composites may be significantly affected by the nature of the interfacial bond between the fiber and the bulk matrix material. Thus microscale details such as the interfacial strength and/or the constitutive response may be extremely important in determining the overall structural behavior. For example the transverse properties of (uni-directional) fiber reinforced composite are strongly influenced by the interface behavior. Also the axial strength and progressive failure in compression of composite structures are greatly affected by interface characteristics. However, there is still a lack of understanding of the relation between the local interface behavior at the microlevel and the overall mechanical behavior and fracture at a macroscopic level. The aim of the investigation was to address this issue from a mechanics perspective. Our attention was limited to the transverse behavior and we use the finite element method to simulate the micromechanical response of the composite and its effect on overall deformation and fracture.

## 2. Accomplishments

The following discussion of research accomplishments consists of two parts. The first of these pertains to the short duration of the research contract with NCCMR, i.e. the sixmonth period from April 1, 1989 to September 30, 1989. This phase of our research laid the foundations for the work which we have continued under contract from ONR. In the second part, we outline some of our research accomplishments on the ONR project which built upon our activities under contract from the National Center for Composite Materials Research.

## 2.1 Research accomplishments contract N00014-86-K-0799, Subcontract No. 89-122

Many investigations of the transverse behavior of composite materials have been carried out using appropriately defined unit cells or composite subdomains. In the majority of these investigations, it has been assumed that the bond between the fiber and matrix is

# UNIVERSITY OF PENNSYLVANIA

#### FINAL REPORT

#### COMPOSITE MATERIAL INTERFACE MECHANICS

GRANTS N00014-86-K-0799 N00014-90-J-1750 N00014-91-J-4044

Principal Investigator: Professor Z. Hashin
Dept. of Mechanical Engineering and Applied Mechanics
University of Pennsylvania
Philadelphia, PA 19104-6315

These grants were funded jointly by the Air Force Office of Scientific Research and by the Office of Naval Research through the National Center for Composite Materials at the University of Illinois, Urbana-Champaign. The effective time period of these grants was March 1, 1989 - September 30, 1991 with a no-cost extension for the period October 1 - December 31, 1991. The work of the principal investigator was done at the Dept. of Mechanical Engineering and Applied Mechanics, University of Pennsylvania.

The major part of the scientific work has been concerned with quantification of the concept of imperfect interface, the modifications of the theory of composite materials for imperfect interface and evaluation of the effect of various kinds of imperfect interface on various properties of various composite materials. In addition a study of the effect of matrix nonlinearity and imperfect interface on the critical compressive stress of unidirectional fiber composites has been performed. The work performed has been published and submitted for publication in 8 papers which are listed below.

- 1. Z.Hashin, "Thermoelastic properties of fiber composites with imperfect interface", Mechanics of Materials, 8, 333-348, (1990).
- 2. Z. Hashin, "Composite materials with viscoelastic interphase", Mechanics of Materials, 11, 135-148, (1991)
- 3. Z. Hashin, "Composite materials with interphase: Thermoelastic and viscoelastic effects", in <u>Inelastic Deformation of Composite Materials</u>, G.J. Dvorak, Ed., Springer Verl., 3-34, (1991).
- 4. Z. Hashin, "Micromechanics aspects of damage in composite materials", in <u>Durability of Composite Materials Systems</u>, A.H. Cardon and G. Verchery, Eds.

### UNIVERSITY OF PENNSYLVANIA

Elsevier Applied Science, 27-45, (1991).

- 5. Z. Hashin, "The spherical inclusion with imperfect interface conditions", J. Appl. Mech., 58, 444-449, (1991).
- 6. Z. Hashin, "Thermoelastic properties of particulate composites with imperfect interface", J.Mech.Physics Solids, 39, 745-762, (1991)
- 7. Z. Hashin, "Extremum principles for elastic heterogeneous media with imperfect interface and their application to bounding of effective moduli", J.Mech.Physics Solids (in press).
- 8. Z. Hashin, "Effect of matrix nonlinearity and imperfect interface on compressive strength of fiber composites", (to be published)

Attached are copies of the first pages of the above listed publications, including abstracts.

In addition the principal investigator has given invited lectures on these subjects at the following places and occasions, during the grant period:

Yale University
University of No.

University of North Carolina (Raleigh) - Distinguished Lecture Seminar

Princeton University

University of Pennsylvania

Arizona State University

Rensselaer Polytechnic Institute

University of Delaware

Free University of Brussels, Belgium

University of Illinois, Urbana

Annual Meeting of the Society of Engineering Science, Eringen Medal acceptance lecture, Ann Arbor, MI, October 1989.

IUTAM Symposium on Inelastic Behavior of Composite Materials, RPI, NY, August 1990

Annual Meeting of the Society of Engineering Science, Santa Fe, NM, 1990

### VIRGINIA POLYTECHNIC INSTITUTE AND STATE UNIVERSITY

Final Technical Report
ONR/URI Research
UI ONR Contract N00014-86-K-0799
UI Subcontract 86-159
URI Annual Review February 5-7, 1990 Urbana, Illinois

#### EXPERIMENTAL STUDIES OF THICK COMPOSITES

Reported by
Daniel Post
Virginia Polytechnic Institute and State University
Engineering Science and Mechanics Department
Blacksburg, Virginia 24061-0219

This report highlights three analyses: (1) residual strains in University of Maryland specimens; (2) shear stress-strain behavior of DTRC thick-walled cylinders; and (3) interlaminar strains at hole boundaries in DTRC plates in compression. Additional analyses of thick-section composites are listed; details are given in our Annual Progress Report, September 1988-9.

Two new initiatives will be undertaken in 1990 under direct ONR Sponsorship. In one, a unique ring—test fixture developed by Hercules Aerospace will be used to apply external pressure, while micromechanical deformations of thick—walled rings are measured by moire interferometry. In the other, a new method for determination of shear stress—strain properties of composites will be investigated.

Research to advance experimental techniques is continuing. The development of Ultra-high sensitivity techniques for deformation measurements in microscopic fields of view is being pursued with vigor.